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PATENT SPECIFICATION

1,075,689

DRAWINGS ATTACHED

1,075,689

Date of Application and filing Complete Specification: July 21, 1965.

No. 31018/65.

Application made in United States of America (No. 384,381) on July 24, 1964.

Complete Specification Published: July 12, 1967.

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Index at acceptance:—B5 B2A2B

Int. Cl.:—D 01 f

COMPLETE SPECIFICATION

Textile Yarn

ERRATUM

SPECIFICATION No. 1,075,689

Page 1, Heading, Application made in United States of America *for* "(No. 384,381)" *read* "(No. 384,831)"

THE PATENT OFFICE
18th December 1967

and increases the weight of the fabric. Fabrication of woven, light-weight fabric with a useful degree of stretch as heretofore been impractically expensive. It is true that synthetic fiber yarns which have artificially induced crimpiness have some stretchability, but this has invariably been either of minor degree and of low recovery power and rate or so limited by twist-liveliness as to have no practical utility in light-weight flat fabrics. Such yarns may have, for example, either mechanically induced crimpiness, heat-set-twist or spontaneous crimpability based on a bicomponent structure of the type more fully described below. Crimping confers bulkiness but little practical stretch; yarns which have been twisted, heat-set and untwisted can yield fabrics having some stretch, but twist-liveliness of such yarns leads to crepiness unless suitably plied to balance the inherent twist-liveliness which severely limits such a process to heavier weight woven fabrics.

[*Price*

This invention provides a novel synthetic composite filament. It more particularly provides a synthetic multi-component filament which is capable of developing a high degree of helical crimp against the restraint imposed by high-thread-count woven structures, which crimp potential is unusually well retained despite application of elongating stress and high temperature. It further provides a synthetic composite filament which increases, rather than decreases, in crimp-frequency potential when heat-set. It still further provides aesthetically pleasing, light-weight, flat fabrics with good stretch, stretch recovery and stretch power comprising said filaments. It also provides a route to flat, continuous-filament fabrics of durable stretchiness without the problems associated with twist-lively yarns.

The stated advantages of this invention are achieved in a helically crimpable composite filament substantially comprising a laterally eccentric assembly of two or more

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We, E. I. Du Pont De Nemours and Company, a company organised under the laws of the State of Delaware, United States of America, of Wilmington, State of Delaware, United States of America, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to self-crimpable synthetic fibers and, more specifically, to self-crimpable bicomponent fibers which have unusual crimp development properties.

It is frequently desirable in applications of synthetic fibers to achieve a degree of stretchability not inherently characteristic of fabrics made therefrom. In the past such stretchability has usually been attained by incorporation of an elastomer, such as natural rubber or a synthetic rubber, into the fabric structure. This alters tactility markedly and increases the weight of the fabric. Fabrication of woven, light-weight fabric with a useful degree of stretch as heretofore been impractically expensive. It is true that synthetic fiber yarns which have artificially induced crimpiness have some stretchability, but this has invariably been either of minor degree and of low recovery power and rate or so limited by twist-liveliness as to have no practical utility in light-weight flat fabrics. Such yarns may have, for example, either mechanically induced crimpiness, heat-set-twist or spontaneous crimpability based on a bicomponent structure of the type more fully described below. Crimping confers bulkiness but little practical stretch; yarns which have been twisted, heat-set and untwisted can yield fabrics having some stretch, but twist-liveliness of such yarns leads to crepiness unless suitably plied to balance the inherent twist-liveliness which severely limits such a process to heavier weight woven fabrics.

Composite bicomponent filaments comprising two or more synthetic components, which differ in their ability to shrink, in eccentric, intimately adhering, coextensive relationship are known in the art. In U.K. Specification No. 805,033, for example, it is shown that two condensation polymer components which differ in shrinkability can be co-spun in either eccentric sheath-core or side-by-side relationship. Such a filament crimps helically when subjected to shrinking conditions in an essentially tensionless state, the number of crimps-per-inch being directly related to difference in shrinkage between components. Such crimp is useful in providing bulk and resilience in fabrics, stuffing materials, etc. Such bicomponent fibers have been severely limited in their ability to crimp against a restraining load such as that encountered in woven fabrics and lose very substantially in their ability to crimp when heat-set to low, total shrinkage.

This invention provides a novel synthetic composite filament. It more particularly provides a synthetic multi-component filament which is capable of developing a high degree of helical crimp against the restraint imposed by high-thread-count woven structures, which crimp potential is unusually well retained despite application of elongating stress and high temperature. It further provides a synthetic composite filament which increases, rather than decreases, in crimp-frequency potential when heat-set. It still further provides aesthetically pleasing, light-weight, flat fabrics with good stretch, stretch recovery and stretch power comprising said filaments. It also provides a route to flat, continuous-filament fabrics of durable stretchiness without the problems associated with twist-lively yarns.

The stated advantages of this invention are achieved in a helically crimpable composite filament substantially comprising a laterally eccentric assembly of two or more

synthetic polymeric components at least one of which is a polyester partly crystalline in stable conformation 90% or less the length of the fully extended molecular conformation, said one component generally assuming a position on the inside of the crimp helices formed when the fiber crimps, and in yarns and fabrics made therefrom. The term "stable" means that the polymer retains its crystalline conformation under all conditions which do not melt the polymer.

In a preferred embodiment the components are eccentrically coextensive throughout the length of all filaments, and are present in a substantially constant ratio in the filament cross section. In another embodiment each filament has a substantially constant ratio of the components arranged eccentrically throughout its length, but the ratio of the components present is varied from filament-to-filament. In still another embodiment, the components vary in their cross sectional ratio both filament-to-filament and along a given filament. Each of these embodiments has characteristics which are peculiar thereto, but all have in common the advantages of practical crimpability against restraining load imposed by woven fabrics made therefrom, good stretchability and moderate-to-high-power recovery from stretch.

Both components may comprise polyesters having a non-extended crystalline conformation or even "non-extended" polyesters having the same chemical composition if care is taken in selection of other polymer characteristics. A difference in molecular weight between components or use of a homopolymer for one component and a copolymer for the other, as examples, can provide the necessary differential in shrinkability and, thereby, spontaneous crimpability. It is preferred, however, that the second component, that is, the component which is located on the outside of the helix when the fiber is crimped, comprises a polymer which is more extended in its crystalline conformation than is the polymer of the first component. Even more desirable products result, generally, if the second component crystallizes in a conformation in which the crystalline repeat-distance is 95% or more that of the fully extended chemical repeat distance.

The crystalline conformation of the component which is inside of the helix of the crimped, composite filament, as has been mentioned above, must be non-extended to the extent that the crystalline repeat-distance is 90% or less that of the fully extended chemical repeat-distance. Although the reasons for this requirement are not fully established, and the invention should not be limited by a theoretical consideration, the discussion which follows may assist in understanding the nature of the invention.

Spontaneous crimping of a bicomponent fiber is the result of differential length changes between components which arise from a difference between components in retraction-from-draw, shrinkage, or both; these, in turn, are due to molecular disorientation involving bond rotation leading to molecular conformation changes. The forces leading to such changes arise from directionally unbalanced kinetic energy of bond rotation as a result of molecular orientation during drawing. There is essentially no phenomenological distinction between retraction-from-draw and shrinkage. The distinction is found only in the environment required for the effect to occur. Retraction-from-draw is fully analogous to retraction from stretch in an elastomer in that it occurs at room temperature. The crimp resulting from a differential in retraction-from-draw, therefore, will develop upon unwinding a yarn from its package and allowing it to relax. Shrinkage refers to that contraction in length which occurs when an oriented fiber is for the first time heated under conditions of low restraint (or none) to a given temperature above ambient temperature. In general, both shrinkage and retraction-from-draw are enhanced by higher orientation in the amorphous zones of the fiber. Stable crystallites in the fiber cannot contribute to shrinkage, since there is no opportunity for bond rotation in a zone where, by definition, it is prevented by intermolecular order. The potential of a fiber component to shrink is affected, however, by changes in the degree of crystallinity. The direction of this effect, that is, to increase or to decrease shrinkage, is determined by the characteristic crystalline conformation of the polymeric component. If the polymer crystallizes in an extended conformation, (as do most polyesters), that is, one in which the length of the crystalline repeat-unit closely approaches that of the fully extended chemical repeat-unit, a unit added will increase the length of the crystallite more than its removal reduces the length of the amorphous region; thus the partially crystalline "extended-type" component loses amorphous orientation (and shrinkage) as it continues to crystallize. If, on the other hand, the added chemical repeat-unit, being of the non-extended type, increases the length of the crystallite less than its removal reduces the length of the amorphous region, an increase in amorphous orientation results. Thus, further crystallization of such non-extended polymer components results in higher tendency to shrink.

From the above discussion, it follows that the unusual properties of the fiber of this invention are fully realized only if the overall orientation of the high-shrinkage component in the uncrimped fiber is greater than its orientation in its crystalline conformation. Thus it it is desirable that this,

the inner component, be the more highly oriented member.

Orientation of synthetic fiber may be accomplished in either or both of two ways: (1) by withdrawing the solidifying filament from the spinneret at a rate higher than its extrusion velocity and (2) mechanical stretching of the solidified filament. In a bicomponent fiber, it is obvious that the mechanical stretching step imparts the same draw ratio to both components and is therefore not highly effectual in establishing an advantage in orientation for the high-shrinkage component. To ensure that the advantage in orientation be accomplished, it is desirable that the high-shrinkage component be more highly oriented during spinning. This is conveniently done by using a higher molecular weight (higher melt-viscosity) polymer for the high-shrinkage component. This same effect can be realized, however, by suitable use of melt-viscosity adjuvants in one component, and such process variations are within the scope of this invention.

The use of a polyester which crystallizes in a non-extended conformation as the high-shrinkage component of a bicomponent fiber provides substantial advantages over a combination of polymeric components which shrink unequally due to another difference such as in molecular weight, tensile recovery, glass transition temperature, and the like. While the composite fibers known in the art possess differing degrees of potential shrinkability among components as a result of variations in response of the components to manufacturing conditions employed, in no instance has such difference been found completely retained or, more strikingly, enhanced by stress and/or high temperature, as is the case with the products of this invention.

Maintenance of the critical crimp-potential (differential shrinkage and retraction between components) requires a good degree of geometrical stability intermolecularly in the high-shrinkage component, except for those rearrangements necessary to fiber shrinkage. Otherwise, the potential energy required for crimping may be dissipated by molecular rearrangements to a more stable condition (of lower shinkage). It has been found that some polymers which crystallize in a non-extended conformation fail to meet the requirements of the high-shrinkage component of this invention apparently due to unstable intermolecular geometry. It will be obvious that any intermolecular order instability ("slippage") will result in reduced total orientation whether it occurs in the crystalline or amorphous regions of the structure.

Accordingly and subject to the limitation that at least one component be a polyester partly crystalline in stable conformation not greater than 90% of the length of the fully extended molecular conformation, it is apparent that composite fibers of the present invention can be any of the materials heretofore employed in composite or other fibers. Condensation polymers and copolymers, e.g. polyesters, polyamides and polysulfonamides, and especially those that can be readily melt spun are preferred for the other components. Suitable polymers can be found, for instance, among the fiber-forming polyesters and polyamides which are described in U.K. Specifications Nos. 461,236 and 578,079 and U.S. Patents Nos. 2,071,250, 2,190,770, 2,130,523 and 2,130,948. The polyesters that are preferred for the component exhibiting the critical stable conformation are poly(trimethylene terephthalate), poly(tetramethylene terephthalate), poly(trimethylene dinaphthalate)* and poly(trimethylene bibenzoate). Poly(ethylene terephthalate) is the preferred polyester for the other component, but other polyesters such as the corresponding copolymers of ethylene terephthalate containing sebacic acid, adipic acid or isophthalic acid as well as those containing recurring units derived from glycols with more than four carbon atoms in the chain can be used as well. Preferred polyamides comprise, for example, such polymers as poly(hexamethylene adipamide), poly(hexamethylene sebacamide), poly(epsilon caproamide) and copolymers thereof.

The conformations of a number of polymers in their crystallites have been deduced from X-ray and model data. Table I gives the chemical and crystalline repeat-distances for a number of polymers:

* Trimethylene dinaphthalate throughout the application means trimethyl 2,6 - dinaphthalate.

TABLE 1
Repeat-distances (Å)

Polymer repeat-unit	Chemical	Crystalline	% Extended
ethylene terephthalate	10.9	10.7	98.
trimethylene terephthalate	12.2	9.1	75
tetramethylene terephthalate	13.4	11.7	87
trans-cyclohexanedimethylene terephthalate	14.7	14.2	97
ethylene 2,6-dinaphthalate	13.4	13.1	98
trimethylene dinaphthalate	14.5	11.5	79
trimethylene bibenzoate	16.6	13.3	80
1,3-cyclobutane dimethylene terephthalate	14.3	13.4	94
1,3-cyclobutane dimethylene bibenzoate	18.6	18	97
hexamethylene adipamide	17.4	17.2	99

Determinations of this nature are accomplished as follows: Measurement of the "% Extended" parameter is done as follows (the order of steps A and B is immaterial):

Step A — Measurement of Crystalline Repeat-Distance. A parallel bundle of oriented and partly crystalline fibers is mounted in an X-ray beam with the fiber axis perpendicular to the beam. A flat photographic film is placed in and perpendicular to the X-ray beam at a distance of a mm. from the fiber array on the opposite side from the X-ray source. The film is suitably exposed and developed to show a "fiber pattern" consisting of a family of more-or-less complete hyperbolae with its axis parallel to the fiber axis, i.e., on the so-called meridian. The distance on the film along this line from the primary-beam image to each hyperbola is measured and designated e_n, n being the ordinal number of the layer line counting away from the equator as zero. The diffraction angle u_n is defined as

$$u_n = \tan^{-1} e_n/a.$$

The identity period (Crystalline Repeat) is simply calculated from

$$I.P. = \frac{n \lambda}{\sin u_n}$$

where λ is the wavelength of the X-rays used. The above notation follows G. L. Clark, "Applied X-Rays", McGraw-Hill, New York (1955), p.401. The patterns from various polymers and particular values of e_n, of course, differ.

Step B — Measurement of Chemical Repeat-Distance. A molecular model of the polymer in question is made from a commercially available scale-model kit such as the "Dreiding" kit manufactured by W. Buchi, Flawil, Switzerland. The interatomic bonds along the chain are rotated so as to give the longest straight length along the molecular chain. The distance from any nucleus in the chain to the corresponding nucleus in the next repeat-unit is measured, converted to Angstrom units, and designated the Chemical Repeat of the polymer.

Step C — Calculation of % Extended. The Crystalline Repeat-Distance from Step A (which is in Angstrom units) is divided by the length calculated from measurements in Step B. The result is multiplied by 100 to give the per cent extended.

If the result of Step C is greater than 100%, obviously the crystal repeat-unit consists of more than one chemical repeat-unit. The actual number can sometimes be estimated from geometrical considerations or from a more detailed analysis of the X-ray pattern. Since the crystal repeat must be

an integral number of chemical repeats, assigning one chemical repeat therefore gives the maximum possible extension: if there were two chemical repeats, the per cent extension would be halved, etc.

The art of composite filaments, to which the present invention generally relates, is well developed and reference may be made to techniques already known for application to the present discovery. A partial list of U.S. patents that may be referred to for this purpose includes Breen, 2,987,797; Radow et al, 3,039,174; Breen, 3,038,236; Taylor, 3,038,237; Breen, 2,931,091; and Zimmerman, 3,038,235. In addition, the various spinnerets described in those references as well as the manner of using them may be used in this invention. Others can, of course, be employed and one suitable type is indicated in the attached drawing in which:

Figure 1 is a schematic, transparent view of a spinneret plate of the post-coalescence type;

Figure 2 is a side view of cooperating channels in a spinneret assembly;

Figure 3 is an enlarged view showing the face plate openings of channels of the type of Figure 2; and

Figures 4, 5 and 6 are cross-sections of some of the type filaments that can be produced in this invention.

Filaments are produced with the indicated type spinneret by conventional procedures. Accordingly, and referring to Figures 1, 2 and 3, by means not shown, two melts are separately metered into the two rings of holes designated as 1 and 2, in Fig. 1 in spinneret 4. A sealing means (not shown) prevents mingling of the two melts at the back face 3 of spinneret 4. The two melts flow through individual channels 5 and 6 to the front face 7 of spinneret 4 where they merge into a side-by-side composite filament as they leave the spinneret assembly.

The filament generally is withdrawn from the spinneret at a speed that attenuates the filament, and is thereafter drawn. The conditions applied for drawing the spun multicomponent filaments of this invention may vary in wide limits. In addition to the processes of drawing described in some of the examples given hereinafter, a hot pin may be used, or the yarn may be passed over heated rolls, as additional examples. In general, the precise amount of draw is established by use of feed and drawing rolls which are driven at the appropriate differential in speed, care being taken to assure that the yarn doesn't slip on either. Two methods for assuring positive control of speed which are appropriate for feeding to or withdrawing from a drawing zone are described in U.S. 3,101,990.

Also, the temperatures at which the filaments are drawn may vary in wide limits and depend mostly upon the properties of the single materials forming the composite filament and of the final desired results. As is the case in the production of conventional unitary filaments, the preferred drawing temperatures for the composite filaments of this invention may vary between room temperature and slightly elevated temperatures; for example temperatures of about 100°C or somewhat higher may be used. Since in the present invention combinations of at least two different materials usually are employed, the specific drawing characteristic of each material should be considered in order to obtain best results. Drawing temperatures which are lower than the glass transition temperature (Tg) of the several components may be employed where a separate plasticizing step is provided. Moreover, if desired the drawing and taut heat treatment may be coupled in a continuous process to obtain the desired orientation and crystallization.

The filaments can thereafter be treated by crystallizing under conditions wherein no shrinkage can occur. In other words the crystallization is effected under conditions of tension which equals the forces developed in the filaments during the treatment. Crystallization or length stabilization respectively of many of the fiber-forming high polymeric materials can therefore be accomplished preferably by a heat treatment of the taut filaments. The temperature applied should generally be higher than the apparent minimum crystallization temperature of the higher shrinkage component which is well konwn or can easily be determined for each polymer. A convenient method for determining the apparent minimum crystallization temperature (Ti) is described, e.g., in U.K. Specification No. 695,567. Preferably, however, the apparent minimum crystallization temperature is determined by X-ray diffraction measurements on samples of filaments which have been drawn in cool water to prevent crystallization and which have been subjected to taut heat treatment at progressively increasing temperatures. Filament exposures are suitably made on a Hilger semi-micro-focus diffraction unit using a flat plate Norelco micro-camera similar in design to that described by Fankuchen and Mark, J. Applied Physics 15,364 (1944). The degree of crystallinity may be judged by direct examination of the diffraction pattern or from radial densitometer traces along the equator of the X-ray diagram. Such a trace will show distinct peaks of fibers having a well developed crystalline structure whereas with an amorphous structure or with very low degrees of crystallinity the peaks cannot be resolved. The apparent minimum crystallization temperature by this method is the minimum temperature of heat treatment at which a definite crystalline structure is detectable

from direct examination of the X-ray diagram or at which distinct peaks are observable in the densitometer trace.

It is the important characteristic of those polyesters having the crystalline repeat distance 90% or less than the chemical repeat distance that largely contributes the enhanced and highly unusual properties of composite fibers in the present invention. For the composite fibers known heretofore, heat treatment at constant length tended to destroy, or at least lessen, shrinkage characteristics. That happens in the present invention also, but only with respect to the components crystallizing in the fully (or more fully) extended form. For the indicated special polyesters, such heat treatment enhances the shrinkage characteristic. Consequently, with a preferred combination in the composite assembly, the shrinkage differential of the two components will ordinarily be greater after the taut heat treatment than before.

Therefore, any treatment which meets the requirement of effecting crystallization of the low shrinkage component may be used in annealing fibers in this invention. A taut heat treatment at a temperature high enough and for a time long enough to provide crystallization of the low shrinkage polymeric component is preferred. The temperatures applied in this heat treatment will generally be higher than the apparent minimum crystallization temperature (Ti) of the component which is to be crystallized, which is defined as the lowest temperature at which the fiber may be treated to produce a substantial degree of crystallinity in its structure. Only a short period of time at the annealing temperature is needed, for example only a fraction of a second. Extended annealing times are not deleterious, but neither or they normally economic, so that they are not used. In the examples given hereinafter, the term "annealing" indicates that the yarn was exposed to the indicated temperature for about 0.1 seconds or longer while held at constant length.

The filaments as produced upon taut annealing may be used as such and crimp may be developed in the ultimate product. Alternatively, crimp may be developed first, and the crimped product then used. Any conventional hot relaxing step now known in the art may be employed to develop the characteristic helical crimp.

In addition to the side-by-side species produced in the exemplified spinneret, it might sometimes be desirable to spin a bundle of filaments which comprises composite filaments containing the components in various ratios through one and the same spinneret. An example is a bundle of two-component composite filaments which comprise filaments consisting of 20% by weight of the higher shrinkage component and 80% by weight of the other, a 30%/70% ratio, a 40%/60% ratio and a 50%/50% ratio, respectively. Such filament bundles containing composite filaments with various ratios of components can very conveniently be produced by utilizing the spinneret which is shown in Figures 6 and 7 of the U.S. patent to Breen 3,118,011. As a generality and without regard to the type composite fiber being produced, the non-extended component usually comprises 20 to 80% of the composite and the other components comprise the remainder. The denier of the resulting product will be that usually produced in this general art, and is not of significance to the invention.

The invention will be described further in conjunction with a series of examples. In the examples, except as otherwise indicated, the terms employed in evaluating polymers and fibers have the following meanings:

Relative Viscosity refers to the ratio of the viscosity of a 10% solution of the polymer in a mixture of 10 parts of phenol and 7 parts of 2,4,6 - trichlorophenol (by weight) to the viscosity of the solvent itself, both measured at 25°C and expressed in the same units.

Intrinsic Viscosity is defined as the limit of the fraction

$$\frac{\ln(r)}{c}$$

as concentration c approaches zero, where r is the relative viscosity as defined above, except that relative viscosity is measured at several concentrations to facilitate extrapolation to zero concentration, and the solvent employed in this measurement is a mixture of three parts of methylene chloride and one part of trifluoroacetic acid (by weight). A more detailed discussion of methods of measuring relative and intrinsic viscosity is given in *Preparative Methods of Polymer Chemistry*, Sorenson & Campbell, Interscience, 1961.

Skein Shrinkage is determined by the following procedure:

1. From the known denier of the yarn, calculate the number of turns of a skein reel required to achieve a denier of 1500 (167 Tex) (the circumference of the reel may be any convenient length), using the formula

$$T = \frac{1500}{d}$$

where T designates turns on the skein reel

and d is denier of the yarn; round off to the nearest even number of turns. Prepare and label a skein from each yarn to be tested. It will be obvious that such a skein must be considered as 3000 denier (334 Tex) when loaded as a loop.

2. Hang the skein and apply a 300-gm. weight at the bottom of the loop. Exercise gently four times. Wait 15 seconds and measure initial length of the skein (L_o).

3. Replace the 300-gm. weight with a 4.5-gm. weight and immerse the skein in boiling water for 15 minutes. Remove from bath.

4. Dry the skein overnight under the 4.5-gm. load. Exercise and measure crimped length (L_c) as in 2 above.

5. Reapply the 300-gm. load, exercise and measure extended skein length as in 2, above (L_e).

6. Calculate skein shrinkage by the formula

$$SS = \frac{(L_o - L_c)\,100}{L_o}$$

Crimp Development: Calculate from data obtained in the Skein Shrinkage procedure by the formula

$$CD = \frac{(L_e - L_c)\,100}{L_e}$$

Crimp Elongation: Determine by the procedure employed in Skein Shrinkage with the addition of one more step: after measurement of L_c (Step 5), the 300-gm. load is replaced by the 4.5-gm load and a second measurement of recovered length under the smaller load is made as in Step 2 of that procedure (L_r). Crimp Elongation is calculated by the formula

$$CE = \frac{(L_e - L_r)\,100}{L_r}$$

EXAMPLE I

This Example illustrates batch preparation of poly(trimethylene terephthalate), coded herein PPT polymer.

Catalyst for this preparation is prepared as follows: Sodium (2.5 gms) is dissolved in 300 ml. of n-butanol. Tetrabutyl titanate (37 gms) is then added and the mixture diluted to 500 ml. with n-butanol.

Dimethyl terephthalate (5.45 kg.) and trimethylene glycol (4.54 kg.) are heated for 100 minutes at 225°C in the presence of 99 cc. of the stock catalyst solution described above. During this time, 1.8 kg. of methanol are removed. The resulting low molecular weight material, to which a small amount of titanium dioxide has been added as a delusterant, is heated further, with stirring, for 6 hours at 250°C under an absolute pressure of 0.4 mm. of mercury during which time the glycol evolved during further condensation is removed. The resulting polymer has an intrinsic viscosity of 0.71.

Poly)trimethylene terephthalate) can be made in a variety of ways, many of which are well-known in the art. Since the process detail employed in its manufacture is not critical to the utility of PPT polymer in practice of this invention, polymer made by several processes has been employed herein indiscriminately.

EXAMPLE II

This Example illustrates a means of enhancing the molecular weight, as evidenced by an increase in intrinsic viscosity, of a polymer such as that prepared in Example I.

PPT polymer of less than 1.0 intrinsic viscosity is cut twice to pass through a 1/8 inch mesh screen, dried 6 hours at 150°C and placed in a vessel through which inert gas is passed. The inert gas and vessel are heated to 180° for two hours, then to 200°C for an additional 12 hours. The polymer and apparatus are then cooled and the polymer removed. The intrinsic viscosity of the finished polymer is 1.29. A second batch of this polymer is prepared and found to have an intrinsic viscosity of 1.36.

Polyethylene terephthalate (PET polymer) may be prepared by any of a variety of procedures known in the art, such as one of the methods taught in U.K. Specification No. 578,079, or one of the methods described in U.K. Specification No. 868,496. While each of these methods may have merit over another in some respect such as in the production of a whiter polymer or in improved space-time yield, these differences are not critical to the purpose of this invention. PET polymer employed in these examples has accordingly been derived from several procedures as dictated only by convenience and availability.

EXAMPLE III

Examples III and IV illustrate a preferred process for preparation of a bicomponent fiber of this invention.

The blended PPT polymers of Example II, and a PET polymer of 18 relative viscosity are cospun at 280°C from a 34-hole spinneret similar to that described in Figure 14 of U.K. Specification No. 1,016,861; that is a pre-coalescence spinneret, that is, one in which the two melts are brought together just behind each extrusion capillary. The filaments are withdrawn from the spinneret

at 468 ypm, drawn 4.70 (that is, to 470% of their original length) at 93°C, and annealed at 170°C. The 68 denier (7.6 Tex) yarn has a skein shrinkage of 12%, crimp development of 55% and crimp elongation of 106%.

EXAMPLE IV

A 34-filament bicomponent yarn is prepared from a PPT polymer in 1.2 intrinsic viscosity and a PET polymer of 20 RV in a ratio of 40/60, by cospinning at 280°C from the post-coalescing spinneret shown in the drawing. The filaments are withdrawn from the spinneret and wound up at 820 yards per minute. The spun yarn is drawn 3.982x at 107°C and annealed at 150°C. Properties of the yarn are: denier 63, tenacity 3.2 gpd, elongation to break 11%, skein shrinkage 16%, crimp development 52%, crimp elongation 95%. Contrary to the procedure generally used herein, the latter three measurements are made at 1.4 mg/den. restraint rather than the standard 1.5 mg/den. restraint. (See procedures for these measurements above in which a 4.2 gm. weight would be used wherever a 4.5 gm. weight is specified.) Another sample of the spun yarn drawn 3.982x at 107°C is annealed at 175°C. The yarn has a skein shrinkage of 13%, a crimp development of 58%, and a crimp elongation of 106% (the last three measurements also being made at 1.4 mg/den. restraint).

EXAMPLE V

This example illustrates suitability of a copolymer as the outer component of the composite fiber of this invention.

A PPT polymer of 1.14 intrinsic viscosity and a copolymer of 98% PET and 2% ethylene sodium sulfoisophthalate of 15.5 relative viscosity, prepared by the general procedure of U.K. Specification No. 868,496 are employed in this example.

The two polymers are melted and simultaneously extruded at 288°C as in Example III. The 34 composite filaments are withdrawn at 700 yarns per minute, drawn to 365% of their original length at 94°C in a water bath substantially as described in U.S. patent 3,091,805 and annealed at 190°C at constant length. Properties of the 70-denier (7.8 Tex) yarn are: tenacity 3.5 gpd, elongation to break 16%, skein shrinkage 8%, crimp development 55%, and crimp elongation 103%.

EXAMPLE VI

This Example illustrates suitability of a copolymer as the inner component.

A 34-filament bicomponent yarn is prepared from a copolymer of PPT with 2 mol per cent sodium sulfoisophthalate of 1.25 intrinsic viscosity and PET polymer of 20 RV in a ratio of 40/60, by cospinning from a post-coalescing spinneret at a temperature of 284°C. The filaments are withdrawn at 1080 yds/min., drawn in an 87°C bath 2.37x (or to 237% of their undrawn length) and annealed at 182°C. The properties of the yarn are: denier 72 (8 Tex), tenacity 1.7 g/d., elongation to break 17%, skein shrinkage 19%, crimp development 46% and crimp elongation 67%.

EXAMPLE VII

This Example illustrates the suitability of alternative polymers in the preparation of fibers of this invention.

In Table 2, the codes employed to represent the polymers have the following meaning:

PPB = poly(trimethylene bibenzoate)

PCHT = poly(trans - cyclohexanedimethylene terephthalate) (Solid-state polymerized in accordance with Ex. II)

PPN = poly(trimethylene 2,6 - dinaphthalate)—using as catalyst a mixture of $Mn(OAc)_2$ and Ti(OBu)4

PET = poly(ethylene terephthalate)

PBT = poly(tetramethylene terephthalate)

PPT = poly(trimethylene terephthalate) (Solid state polymerized in accordance with Example II)

Except for the PET polymers and except as noted above, the polymers of this Example are made in accordance with the procedure employed in Example I. They are all spun from post-coalescence spinnerets. Items a and c are drawn dry substantially as described in U.S. patent 3,101,990, in which process the yarn, lubricated with a textile finish, is passed at a uniform rate to and over a surface heated to the temperature indicated in Table 2 to heat the yarn to the desired drawing temperature and then passed over a snubbing pin where drawing tension is applied by the drawing rolls which withdraws the yarn at the appropriately higher speed to produce the desired degree of draw. Item b is drawn by the process of Example V. Items d and e are drawn in a jet in which steam is introduced at a rate sufficient to open the bundle by turbulence and, thereby, heat each filament uniformly to drawing temperature. As is indicated in the data, some of the fibers are annealed at more than one temperature, illustrating the improved crimp development obtained at higher temperatures of annealing.

TABLE 2

Item	Polymeric Components-viscosity inner	outer	Polymer ratio	Spin temp °C.	Draw Ratio	Draw temp °C.	Anneal temp °C.	Den/fils	Tenacity/ Elong.	Skein Shrinkage	Crimp Develop.	Crimp Elong.
VII-a	PPB .85	PCHT .67	50/50	292	3.45	107	160	71/34	1.3/10	2	24	40
							180	73/34	1.2/7	2	31	64
VII-b	PPN .67	PET 18*	50/50	268	3.28	92	135	99/34	2.4/16	9	17	22
							160	99/34	2.4/17	5	22	30
VII-c	PBT 1.1	PET 20*	40/60	278	4.216	110	none	60/34	3.8/15	15	19	20
						110	125	60/34	4.2/13	16	23	24
						110	150	58/34	4.6/17	13	29	36
						110	175	58/34	4.2/13	11	33	47
						110	200	58/34	4.3/16	9	39	58
VII-d	PPB .85	PET 18*	50/50	282	4.60	100	180	73/34	2.0/9.0	3	15	21
VII-e	PPT 1.34	PCHT .67	50/50	308	3.57	100	180	89/34	1.5/8.0	2	27	45

*Relative Viscosity, all others intrinsic viscosity

Items a, b, d, and e spun at 690 ypm; item c spun at 640 ypm.

EXAMPLE VIII

This Example illustrates further the unusual response of bicomponent fibers of this invention to annealing.

PET polymer of 19 relative viscosity and PPT polymer of 1.42 intrinsic viscosity are cospun at 287°C from a post-coalescence spinneret. The 34 filaments are withdrawn at 700 ypm. The spun yarn is drawn at several draw ratios and annealed, in some cases at several temperatures. Data are summarized in Table 3. It should be noted that Item VIII—c is a PET polymer homofiber which has been twisted, heat-set and untwisted by a commercial means available in the art. It is clearly seen that imposition of a small additional draw and heat-setting on Item VIII—b brings about a measurable improvement in crimp properties, while the crimp of Item VIII—c is essentially destroyed by an identical treatment.

TABLE 3

Item	Draw Ratio	Anneal Temp. °C.	Skein Shrinkage	Crimp Development %	Crimp Elongation %
VIII-a	2.11	none	7.9	6	7
		120	5.3	8	9
		140	4.0	11	12
		160	3.0	12	14
		180	3.7	14	17
VIII-b	2.57	none	13.5	14	16
		180	7.6	35	52
	2.57 × 1.147 = 2.94	180	10.5	46	79
VIII-c	* —	216	4.3	39	63
	* × 1.147	180	6.2	6	7
VIII-d	2.91	none	18.3	18	21
		180	11.6	51	94
VIII-e	3.08	none	18.9	19	22
		120	17.8	22	27
		140	19.6	36	52
		160	15.0	50	86
		180	12.9	53	100
VIII-f	3.20	none	20.5	21	25
		180	14.9	61	130

*Draw ratio not known—approximately 3.2.

The next two Examples illustrate the unsuitability of bicomponent fibers more typical of the art.

EXAMPLE IX

A PET polymer of 22 relative viscosity and an 85/15 copolymer of PET/polyethylene sebacate (PET/PES) of 21.1 relative viscosity are spun from a post-coalescence spinneret at 280°C to 34 side-by-side bicomponent filaments comprising 40% PET/PES and 60% PET. The filaments are withdrawn at 600 yards per minute. They are drawn at 90°C to 460% of their original length.

A portion of the yarn is not annealed at high temperature. A second portion is annealed at 160°C while held at constant length. Table 4 summarizes the yarn properties obtained.

TABLE 4

Property	Annealing Temperature °C. None	160
Denier (Tex)	67(7.5)	67(7.5)
Skein Shrinkage %	22	5
Crimp Development	8.6	0.4
Crimp Elongation	3.8	0.4

It is seen that the crimp properties are not outstanding in this fiber in the absence of annealing, and essentially all tendency to crimp is lost by annealing to reduce the high shrinkage, at a temperature which leads to enhanced crimp characteristics in the fiber of this invention.

EXAMPLE X

Two PET polymers, one of greater than 100 relative viscosity and the other of 21 relative viscosity are cospun at 297°C through a 34 hole spinneret such as indicated in the previous examples. The fibers are withdrawn at 840 ypm.

The spun yarn is drawn at 100°C at the maximum operable draw ratio, established by slowly increasing the draw ratio until the yarn breaks, then restringing at a slightly lower ratio (4.08). Samples of yarn are also prepared at somewhat lower ratios for comparison. Table 5 summarizes the data obtained.

TABLE 5

Item	Draw Ratio	Anneal Temp °C	Skein Shrink %	Crimp* Develop.	Crimp* Elong. %
X—a	4.08	none	12.6	1.2	1.4
		120	8.3	0.7	0.5
		140	5.8	1.3	0.9
		160	3.5	0.5	0.5
		180	3.0	0.9	0.5
X—b	3.52	none	11.6	1.0	1.2
		180	1.7	0.7	0.7
X—c	3.08	none	12.2	1.0	1.2
		180	1.7	1.0	1.1

*Differences indicated in these actual data are insignificant.

It is clearly seen that essentially no crimp is developed by the fiber of this example under the standard conditions for measurement of crimp development.

In another evaluation of some of these yarns at zero loads (vs. the standard 4.5 gm. load employed in all other evaluations), it is seen that a moderate level of crimp is achieved (Table 6). It is apparent that because of its inability to develop crimp under the nominal 1.5 mg/den. (4.5 gms/3000 den) loading employed in the routine test, the fibers of this Example will have no utility for preparation of bulky or elastic woven fabrics. Such a level of restraint, or more, is imposed by the fabric structure. Such bicomponent fibers may confer useful bulk and slight stretch in knitted goods wherein restraint is less.

TABLE 6

Item	Draw Ratio	Anneal Temp. °C.	Skein Shrink %	Zero-Load Crimp Devel.	Zero-Load Crimp Elong. %
X—a	4.08	none	10.3	15.6	10.7
		180	3.0	13.7	8.5
X—c	3.08	none	12.9	6.5	5.8
		180	5.5	18.6	12.5

EXAMPLE XI

This Example illustrates the suitability of PPT polymer for both inner and outer components of a bicomponent fiber of this invention.

Two pairs of PPT polymers, the first (XI—a) having intrinsic viscosities of 1.42 and 0.73, and the second (XI—b) having intrinsic viscosities of 1.4 and 0.8, and cospun at 242°C and 285°C, respectively, into 34 composite filaments using a spinneret such as that employed in earlier examples. Item XI—a is withdrawn from the spinneret at 700 ypm and drawn 3.00X; a portion of the yarn is annealed. Item XI—b is withdrawn from the spinneret at 1003 ypm and drawn 3.1X, portions being annealed at two temperatures while a third is not annealed. Table 7 summarizes the data obtained.

TABLE 7

Item	Draw Ratio	Anneal Temp °C	Skein Shrink %	Crimp Develop.	Crimp Elong.
XI—a	3.00	none	15.7	37.3	55.3
		135	10.0	46.2	81.8
XI—b	3.10	none	15	59	125
		100	13	68	178
		150	8	69	191

The next two Examples disclose alternative processes by means of which the unusual behavior of polyesters having non-extended crystallinity may be exploited in bicomponent fibers. Example XII illustrates preparation of yarn in which the ratio of the two polymeric components varies from filament-to-filament. Example XIII illustrates the utility of such non-extended crystallinity in bicomponent fiber having a random distribution of the two components both filament-to-filament and along any given filament.

EXAMPLE XII

In one experiment, a PPT polymer and a PET polymer are cospun in a pre-coalescence spinneret similar to that employed in Example III, except that a doughnut-shaped meter plate is inserted above the ring of conduits feeding PET polymer to the point of coalescence. The meter plate is carefully aligned so that one hole in the meter plate is centered over each of the 34 PET-melt conduits. As is indicated in Table 8, the meter-plate holes vary in diameter so that the rate of flow of the PET melt toward the coalescence point varies widely. No change is made in the rate of pumping the melts to the spinneret, so that the overall denier and the denier-per-filament remain substantially constant, only the ratio of the two polymers in the individual filaments being variable. (PPT/PET ratio in the bundle as a whole is 40//60.) Spinning conditions and yarn properties are given in Table 8A. Examination of a cross section photomicrograph shows a polymer distribution among the filaments which is substantially in agreement with that calculated from the meter plate orifices (see Table 8).

TABLE 8

Hole Diameter	Number of Holes with this Diam.	% of Total	Calculated Polymer Ratio PET/PPT
5 mil	5	14.7	17/83
6	5	14.7	25/75
7	6	17.6	34/66
8	6	17.6	44/56
9	6	17.6	56/44
10	6	17.5	69/31

In a second experiment, the run is repeated using the same spinneret except that the meter plate is omitted. Spinning conditions and yarn properties for this experiment are also given in Table 8A.

TABLE 8A

	With Meter Plate	Without Meter Plate
PPT intrinsic viscosity	1.41	1.68
PET relative viscosity	18	20
Spinning temperature, °C	275	285
Spinning speed, ypm	717	587
Draw ratio	3.63	4.36
Draw temperature, °C	93	93
Annealing temperature, °C	180	185
Yarn denier	70	71
tenacity, gpd	3.4	4.3
elongation, %	22	24
skein shrinkage, %	11	15
crimp development, %	33	68

The yarns of Table 8A and a commercial PET yarn of the same count which has been twisted, heat-set and untwisted are each doubled and knitted into a 4½"-diameter tubing on a circular knitting machine. The fabrics are scoured in boiling water, dried and evaluated (Results given in Table 8b) as follows:

Bulk: A single thickness of the fabric is laid on a base surface of precise flatness and a glass disc, having a weight of 3 gms/cm² and precisely parallel faces, is laid on the fabric. Exact measurements of the height of the upper face of the disc from the base surface are compared with the height of the upper face of the disc when laid directly on the base surface. The volume of fabric beneath the glass disc is then determined by a simple calculation and compared with the weight of the same fabric area. Bulk is expressed in terms of cubic centimeters per gram.

Stretch and Recovery: Values in Table 8b other than bulk are read from an Instron chart prepared as follows: The knit tubing denier is determined. The cross-head of an Instron is set at 2" separation and the fabric sample inserted. The instrument is run at

2" per minute (100% per minute) separation and the stress recorded on a chart moving at 10" per minute and registering a full scale deflection on application of a strain of 0.005 g./denier. Once full-scale deflection is reached, the cross head motion is immediately reversed and returned to the original 2" separation. From the plots of stress vs. % stretch, *Stretch Recovery* is read as

$$\frac{\text{recovered stretch}}{\text{total stretch}} \times 100$$

Work is the area under the curve and is calculated as

$$\int \text{stress (mg/denier)} \times dl_f \text{ (in)}$$

where dl_f is change in fabric length.

Work Recovery is calculated as

$$\frac{\text{work on recovery cycle}}{\text{work on stretch cycle}} \times 100; \text{ and}$$

Power is the stress (mg./denier) observed at one-half of maximum extension.

TABLE 8B

	Yarn With Meter Plate	Yarn Without Meter Plate	Twist-Heat-Set Untwist PET
Stretch, % (at 5 mg/den. stress)	111	112	75
Stretch Recovery, %	91	90	93
Work, mg.in./denier	4.4	5.6	3.2
Work Recovery, %	41	46	50
Power, mg/denier at 1/2 max. ext.	1.8	2.5	2.0
Bulk, cm^3/g	8.7	4.5	8.7

It can be seen that the yarn with meter plate is nearly equal in stretch properties to that without it, and is clearly superior in bulk. Either fiber of this invention is superior to the commercial fiber in per cent stretch and work, and essentially equivalent in other properties. The fiber made with the meter plate was equivalent to the commercial fiber in bulk.

EXAMPLE XIII

A PPT polymer of 1.29 intrinsic viscosity and PET polymer of 18 RV are melted separately and fed in alternating increments of .043 gms. each to a standard all-screen spinning pack of the type commonly used in spinning homofiber yarns. The mixture is extruded through a spinneret of 100 holes, each 15 mils in diameter and 17 mils in depth, at 280°C and wound up at 1001 yds/min. as two 50 filament yarns. The multifilament yarn is subsequently drawn 3.21x at 100°C and annealed at 170°C. The drawn yarn is found to possess a randomly variable crimp among the individual filaments, and the crimp is accentuated by further heat treatment under low or zero restraint. Microscopic examination of filaments prepared in this manner shows them to have varying distribution of the two polymers along their lengths and across their cross sections, (including a few sections that are composed entirely of one individual polymer).

EXAMPLE XIV

This Example illustrates the importance of relative molecular weight between components to development of the desired crimp properties. In general, it has been found that the higher shrinkage component must have the higher melt viscosity at the temperature of spinning. This is conveniently achieved with a higher molecular weight as indicated by relative or intrinsic viscosities. Within the spinnable range of molecular weights for the pair of components, the greater the difference in molecular weight the better the crimp properties.

A PET polymer having a relative viscosity of 19 is spun to 34 filament yarn with each

of three PPT polymers similarly prepared to have differing intrinsic viscosities as noted. Data are summarized in Table 9. In items XIVa, b and c, a 40/60 ratio of polymers is used and the fibers are annealed at two temperatures, further illustrating the effect of this variable. In another experiment, a PPT polymer of 1.79 intrinsic viscosity is cospun in a 50/50 ratio with each of two PET polymers, having different relative viscosities as noted. The effect of molecular weight ratio on properties for both series of experiments is summarized in Table 9.

TABLE 9

Example	XIV-a		XIV-b		XIV-c		XIV-d	XIV-e
Inner Component Intrinsic Viscosity	PPT 1.09		PPT 1.34		PPT 1.48		PPT 1.79	PPT 1.79
Outer Component Relative Viscosity	PET 19		PET 19		PET 19		PET 20	PET 29
Cross Section Ratio	40/60		40/60		40/60		50/50	50/50
Spin Temp. °C.	288		288		288		295	295
Spin Speed, ypm	544		544		544		850	850
Draw Ratio	4.78		4.78		4.78		3.0	3.0
Draw Temp. °C.	95	93	95		92	92*	92	93
Annealing Temp. °C.	195	140	195	140	195	140	185	185
Denier/(Tex)	70(7.8)*	70(7.8)*	71(7.9)	70(7.9)	70(7.8)	70(7.8)*	73(8.1)	68(7.6)
Tenacity/Elongation	4.3/21	3.9/21	4.5/20	3.5/18	4.4/21	4.1/20	2.9/21	3.8/20
Crimp Development, %	35	9	45	20	58	29	64	43
Crimp Elongation, 5%	51	10	74	24	105	38	182	81
Skein Shrinkage, %	10	15	12	17	16	20	15	13

*Estimated value.

EXAMPLE XV

This example illustrates performance in fabric found typical of the fiber of this invention.

Two bicomponent fibers, XV—a and XV—b, prepared from PPT of 1.4 intrinsic viscosity and PET of 21 and 20 relative viscosities, respectively, and the fiber of Example III (XV—d) are processed into plain taffeta fabrics. Table 10 summarizes the data obtained. A plain-weave fabric, XV—c, made of a PET yarn which had been twisted, heat-set and untwisted by a commercially available process is included for comparison.

It is apparent that the elongation, or stretch, and stretch-power of fabrics from the fibers of this invention (XV a, b, d) are much higher than those of the fabric of the prior-art fiber. This improvement in elongation was obtained without sacrifice in recovery from maximum stretch. Furthermore fabrics XV a, b, d, all had a smooth, flat surface similar to normal taffeta or broadcloth fabrics, but the prior-art fabric had a fine surface pucker resembling a crepe. Fabric XV—c could have been heat-set at a narrower width to give a higher finished warp count and increased fabric elongation, but the fabric surface in this case would have been so badly puckered and deformed as to be totally unacceptable for textile uses.

TABLE 10

Item	Spin Speed YPM	Spin Temp. °C.	Draw Ratio	Draw Temp. °C.	Anneal Temp. °C.	Den/fil.	Skein Skg. %	Crimp Dev.	Crimp Elong. %
XV-a	690	268	3.70	90	190**	71/34	15	55	98
XV-b	724	268	3.53	92**	187	70/34	13	62	123
XV-c	—	—	—	—	—	70/34(Warp) 70/54(Fill)		—	—
XV-d	468	280	4.7	93	170	68/34	12	55	106

	Loom Const'n WXF	Finished Const'n WXF	% Recovery in 1 minute from Max. Stretch*	% Elong. 2 lbs/in load	Power at 15% Elong. lbs/inch	Power at 50% of total stretch lbs/in of fabric width
XV-a	104 × 70	172 × 79	99.5	36.8	.99	1.40
XV-b	104 × 70	176 × 79	99.5	40.8	.75	1.30
XV-c	96 × 68	110 × 76	99.9	15.7	—	.62
XV-d	104 × 70	164 × 84	99.7	30	1.34	1.34

*Maximum stretch is taken as that extension resulting from 2 lbs/inch load
**Estimated value.

EXAMPLE XVI

This example illustrates the use of a co-spun antistatic material in the practice of this invention.

PPT polymer (93.2 parts) of 1.53 intrinsic viscosity, in the form of flake gound to pass a $\frac{1}{8}$"-mesh screen is blended with six parts of finely divided polyethylene glycol of 20,000 molecular weight and 0.8 parts of a commercial polyphenol antioxidant by tumbling the three materials together. This physical mixture is designated PPT—A. Mixture PET—A, comprising 93.2% PET polymer of 26 relative viscosity, 6% of the same polyethylene glycol and 0.8% of the same antioxidant, is prepared in a parallel manner. In the data given hereinafter, PPT and PET indicate the foregoing polyester polymers free from those additives.

Using the process of Example XII, two 34-filament bicomponent yarns are prepared with a non-uniform ratio of components filament-to-filament, using the foregoing polymer compositions as the two components, and drawing to varying extents. These yarns are knitted on a $4\frac{1}{2}$" diameter circular knitting machine. The resulting fabrics are scoured in boiling water, dried and evaluated for bulk properties. These variable-ratio products and a similar product having no co-spun antistat are identified in Table 11 opposite "Fiber Composition" by the prefix "V—".

Two more products are prepared by the procedure of Example III to result in a 40/60 ratio of PPT component to PET component, in one instance employing PPT/PET—A and in the second instance using PPT—A/PET—A. The evaluation data obtained on these preparations are summarized in Table 11.

TABLE 11

Fibre Composition	V—PPT—A/ PET	V—PPT—A/ PET—A	V—PPT/PET	PPT/PET—A	PPT—A/PET—A
Draw Ratio	4.0	3.9	4.1	4.9	4.2
Skein Shrinkage (%)	11	11	12	11	11
Crimp Elongation (%)	15	27	63	154	185
Crimp Development (%)	11	19	33	59	63
Tenacity (gms./den.)	2.8	3.1	3.4	4.3	4.0
Elongation (%)	17	33	24	19	25
Initial Modulus (gms./den.)	19	36	21	52	44
Stretch—(%)	124	118	148		
Recovery (%)	94	94	97		
Work (mg-inches/den.)	4.0	4.1	4.7		
Work Recovery (%)	50	47	51		
Power (mg/den.)	1.4	1.5	1.4		
Bulk (cc/gm.)	6.4	6.6	6.4		
Static Propensity*					
Initial Charge (KV)	3.7	4.5	—	3.3	4.6
Discharge: t 1/2 (sec.)	0.5	0.5	—	2.0	0.5
t_{600} (sec.)	7	2	—	83	24

*Initial charge developed in 420 seconds at the center of a 3" × 6" (76 × 152 mm.) strip of fabric to the ends of which is impressed a potential of 5000 volts.

Discharge—t 1/2—time in seconds for the center portion of the fabric to drop to 1/2 of its charged potential after the two clamps are grounded.

t_{500}—time in seconds for the center portion of the fabric to drop to 500 volts after the clamps are grounded.

It is apparent from the data in Table 11 that the valuabe properties of the filament of this invention are not impaired materially by the presence of a cospun antistat such as polyethylene glycol.

From the foregoing discussion, examples and data, the unique results attained in accordance with the present invention in composite fiber assemblies in which at least one component is a polyester partly crystalline in a non-extended conformation 90% or less of the chemical repeat are apparent.

It should be understood that the extent of crystallinity in composite filaments of this invention is generally that which is characteristics of composite filaments known heretofore. The term "partly" as used in the definition of the invention is present merely to eliminate from the scope of the invention the limiting situation of complete crystallinity wherein shrinkage would disappear. The critical limitation in this invention is the character of the polyester that must be present in all of the claimed composite filaments, namely its crystallites being in stable conformation that is 90% or less of the length of the fully extended molecular conformation. When crystallinity is present in these composite filaments, that limitation is met because it is a physical or structural characteristic of the special polyesters that must be present. When that limitation is met, the unusual responses demonstrated in the case result. Hence the scope of the crystallinity has a minimum level of only the presence of some crystallinity, and a maximum level of any amount short of complete crystallinity so that shrinkage is possible. Similarly, the molecular weight of the polymers used may vary widely and generally will be in the range conventionally employed in the synthetic polymer art.

In the practice of this invention, the additives normally employed in the manufacture of synthetic fibers may be used and are substantially without adverse effect on properties of the fiber obtained. It is possible, as examples, to add antistatic agents, delusterants, fluorescent brighteners, dyes, pigments, surface rougheners and the like to one or both components within reasonably wide limits without adversely influencing differential shrinkage, crimp, stretch or stretch-recovery appreciably. Addition of topical finishes may also be practiced.

This invention is applicable to the production of fiber of any cross-sectional shape. Those which have been employed and found satisfactory include, for example, round, oval, ribbon, double round and trilobal.

Although this invention has been largely exemplified in the preferred side-by-side bicomponent structures, sheath-core structures such as those described in U.K. Specification N . 805,033 and other bicomponent shapes are within its scope. It is known that the sheath-core arrangement of components is particularly useful for preparation of bicomponent fibers from components which have such poor adhesion as to split apart when spun in a side-by-side arrangement.

While the exemplified development of crimp by shrinking has exclusively involved treatment in hot or boiling water in this specification, it is recognized that operative alternatives exist which also would be within the scope of this invention. Treatment with a transitory plasticizer, for example, can lower the glass transition temperature of a polymer sufficiently to accomplish the necessary shrinkage for crimping at a temperature substantially below the boiling point of water; it is conceivable that such a treatment could shrink the fiber at room temperature. Further, polymers vary widely in glass transition temperature, and treatment at temperatures substantially above 100°C. may be necessary to shrink fiber made for the high glass-transition-temperature components.

Discussion of utility of the novel filaments of this invention has emphasized light-weight stretch fabrics since use in this type fabric highlights the novel combination of properties offered by these filaments. It is not intended, however, that utility should be restricted to such uses. As suggested in Examples XII, the bulk afforded by the spontaneously crimpable filaments of this invention is useful in a variety of fabrics where bulk and tactility are emphasized rather than stretch and power. The filaments of this invention are also useful as replacement for elastomeric or twisted and heat-set filaments in yarn structures produced by the core spinning processes illustrated in U.S. Patents 2,777,310 and 2,880,566, as examples.

In staple form, the fibers of this invention contribute to improved bulk and stretch in knitwear as well as in woven fabrics. In those cases, where the tactility of fabrics derived from staple yarns is preferred, the fiber of this invention offers new versatility of styling and advantageous functionality. Best processability in the available textile operations for conversion of staple to yarns will be obtained by minimizing crimp development prior to the yarn or fabric stage by use of minimum temperatures in all steps of manufacture so as to minimize early crystallization of the non-extended component.

The excellent stretch and power coupled with broad versatility of the filaments of this invention admirably equip them for use in a wide range of fabrics and end uses. Among the suitable end uses are upholstery, slip covers, carpets, hosiery, half-hose or socks, support hose, ski pants, leotards, boxer shorts, swim wear, sweaters, undergarments where support or bulk is needed, lingerie, brassieres,

girdles, men's suitings (for better fit, wrinkle recovery), etc. Suitable fabric types include wovens, knits and warp-knits as well as non-woven, or felt-like, fabrics, especially where durability, tactile and visual aesthetics, high power of recovery from stretch and simplicity and economy of fabrication are desirable.

It will be apparent to one skilled in synthetic fiber art that other combinations of polymers may meet the requirement of this invention which is that at least one component must be capable of partial, stable crystallization in a non-extended conformation, that is in which the crystalline repeat-distance is 90% or less that of the chemical repeat-distance of the species, said component being higher in shrinkability at moderately elevated temperature than the other component such that said component is located on the inside of the crimp helix in the crimped bicomponent fiber. Bicomponent fibers meeting this requirement, have, without exception, provided the unique performance advantages characteristic of the fibres of this invention.

WHAT WE CLAIM IS:—

1. A helically crimpable composite filament comprising a laterally eccentric assembly of at least two synthetic polymeric components at least the first of which is a polyester partly crystalline in stable conformation that does not exceed 90 per cent of the length of the fully extended molecular conformation of the said polyester, the remaining component or components having a lower shrinkage than the first polyester component, the said polyester being adapted to assume generally a position on the inside of crimp helices formed when the assembly crimps.

2. A filament in accordance with Claim 1 in which the said polyester is poly(trimethylene terephthalate), poly(trimethylene dinaphthalate) or poly(trimethylene bibenzoate).

3. A filament in accordance with Claim 1 or 2 in which the assembly contains a second component which has a crystalline conformation more closely approaching its fully extended molecular conformation than the first component.

4. A filament in accordance with Claim 3 in which the second component has a stable conformation that is at least 95 per cent of the length of the fully extended molecular conformation of the second component.

5. A filament in accordance with Claim 4 in which the second component is poly-(ethylene terephthalate).

6. A filament in accordance with Claim 1, substantially as hereinbefore described in any of the Examples III to VIII, XI to XIV and XVI.

7. A helically crimped composite filament comprising a filament as claimed in any one of Claims 1 to 6 which has been treated in such a manner as to impart a helically crimped configuration to it.

8. A filament in accordance with Claim 7, substantially as hereinbefore described in any of the Examples III to VIII, XI to XIV and XVI.

9. A fabric comprising filaments in accordance with any of Claims 1 to 8.

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Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.
—1967. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2,
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1075689 COMPLETE SPECIFICATION
1 SHEET *This drawing is a reproduction of the Original on a reduced scale*

Fig. 1.

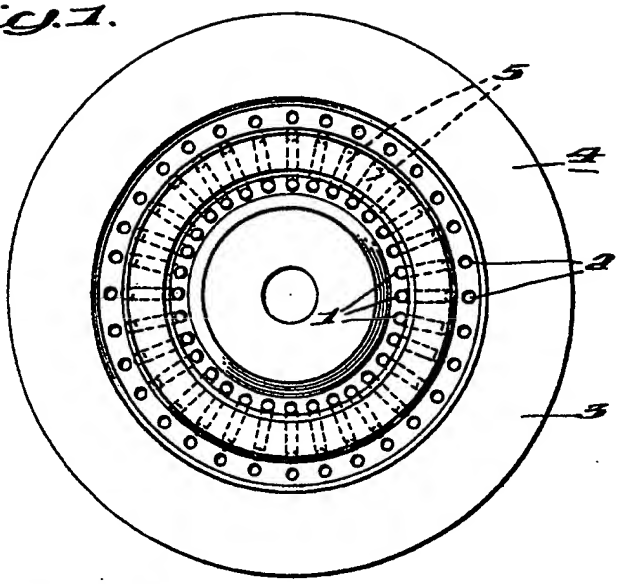

Fig. 2.

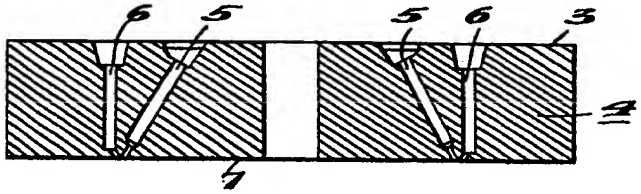

Fig. 3.

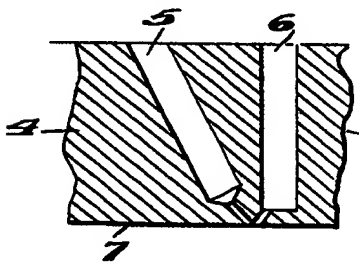

Fig. 4.

Fig. 5.

Fig. 6.